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QUANTITATIVE EVALUATION OF THE RELATIVE EFFECTIVENESS OF VARIOUS METHODS FOR THE ANALYSIS OF ASBESTOS IN SETTLED DUST

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Summary

The presence of asbestos in settled dust in occupied buildings has been a long-standing concern due to potential exposure to asbestos as a result of re-entrainment of the asbestos fibers in the dust. Over the past several years, numerous methods have been explored for sampling settled dust and quantifying the level of asbestos fibers in the dust. After several years of development, three methods for the analysis of asbestos in settled dust are near completion by ASTM. The three methods are:

- Standard Test Method for Microvacuum Sampling and Indirect Analysis of Dust by Transmission Electron Microscopy for Asbestos Structure Number Concentrations
- Standard Test Method for Microvacuum Sampling and Indirect Analysis of Dust by Transmission Electron Microscopy for Asbestos Mass Concentration
- Standard Test Method for Passive Sampling and Indirect Analysis of Dust Fall for Asbestos by Transmission Electron Microscopy

The U.S. EPA Atmospheric Research and Exposure Assessment Laboratory (AREAL) was interested in obtaining an objective assessment of the relative effectiveness of these three methods (plus a new method for direct analysis of dust) and an individual evaluation of the practicality of each method. Research Triangle Institute, under contract to the U.S. EPA, is conducting an evaluation of the three methods to characterize their precision, accuracy, and relative effectiveness in quantifying the presence of asbestos in settled dust.

Sample Generation and Collection

In order to evaluate the various techniques for dust analysis, it was decided to use laboratory-created samples rather than real-world samples. The advantages to using laboratory-created samples include:

- control of asbestos concentration
- control of non-asbestos matrix material composition
- homogeneity of asbestos dispersion within the dust
- control of dust emission source and emission rate
- control of amount of dust deposited on surface

Samples were generated by dispersing an asbestos/matrix mixture in an environmental chamber using a 10 LPM pump and an insufflator filled with approximately 7 grams of dust. An initial test of deposition homogeneity (mass/area) was conducted to verify the adequacy of the system for dust generation. Several dust mixtures were used, incorporating various asbestos concentrations, various matrix materials, and various deposition amounts. Sampling locations were delineated by superimposing a grid on the environmental chamber lower surface. Each grid location was sampled by one of six techniques (microvacuum sampling, passive sampling, passive MCE sampling, direct tape lift, post-it tape lift, and wipe sampling).

Sample Preparation

Samples were prepared primarily by indirect preparation techniques, as specified in the individual analytical methods. Indirect preparation of dust samples involves dispersing the collected material into an aqueous suspension, filtering various aliquots of the suspension onto filter membranes, and then preparing the resultant filters by the direct preparation technique. Direct preparation of filters involves etching of the filter surface by oxygen plasma, evaporation of a carbon layer on the sample surface, and dissolution of the membrane material. Samples are then ready for direct examination by transmission electron microscopy (TEM).

Samples prepared for gravimetric analysis had an additional step of ashing of the filtered material. Wipe samples required ashing of the collection filter before suspension in water. Direct tape lift samples were simply carbon-coated, floated on water to dissolve the matrix material, and deposited on grids. Post-it samples and tape lift samples were directly mounted and carbon-coated for SEM examination.

Sample Analysis

Samples were analyzed by TEM, with individual fiber identity confirmed by fiber morphology, energy-dispersive x-ray spectroscopy, and selected-area electron diffraction. Samples analyzed by the "microvac structure number" method and the "passive dust fall" method were assessed for asbestos structure concentration, as were the wipe samples. Samples analyzed by the "microvac mass concentration" method were assessed for asbestos structure mass, and as a check against the other methods, were also assessed for asbestos structure concentration. Because the direct tape lift samples were not appropriately loaded for TEM analysis, both the tape lift and post-it samples were analyzed qualitatively by SEM.

Results and Discussion

Intersample variation within groups of samples representing each individual method was determined to provide a measure of method precision. Relative accuracy was determined by comparing the results for each method with the relative amount of asbestos deposited in the environmental chamber (asbestos concentration and/or amount of dust deposited). The relative

effectiveness of each method was evaluated by comparing results obtained by the three methods from analyses of samples collected from identical surfaces.

Conclusions

The following conclusions were reached as a result of this study:

The environmental chamber was an effective method for dust dispersion and collection. Dust dispersion was homogenous throughout the chamber, and the chamber was ideal for all types of sample collection.

The microvac structure count method appears to be an effective method of dust collection, preparation, and analysis. Collection of samples is easy and relatively foolproof, and the equipment is portable and widely available. Sample preparation is fairly straightforward, and results show that intersample variability on identical samples is low. The method tracks variable sample asbestos concentration quite well.

The passive method also appears to be an effective method of sample collection, preparation, and analysis. Collection of samples is extremely easy, and can be conducted with a variety of airtight vessels. Sample preparation is straightforward, and intersample variability on identical samples is low. The method tracks variable asbestos concentration quite well.

The microvac gravimetric method is identical to the microvac structure count method up to the point of sample preparation. There the methods diverge. The gravimetric method has a considerably more complex sample preparation system due to the introduction of tared gravimetry and sample ashing. The analysis stage is also very complex due to individual structure measurement and mass calculation. Results obtained on standardized samples showed substantial variability due to the effects of individual large structures. As a consequence of the large structure effects, the analyst must accept the large variability inherent with sample analysis or analyze samples for potentially prohibitive time periods in order to reduce the large structure effect to a tolerable level.

The direct tape lift method (a subsection of the "direct analysis" method) evaluation utilized a tape-like device to remove dust from the sampling surface. The resultant sample was far too heavily loaded for TEM analysis, and would be unsuitable for all but the cleanest sampling surfaces. Analysis by SEM, however, showed that the both the post-it and tape lift samples nicely retained asbestos/matrix spatial relationships, and allow direct examination of the asbestos as it occurs on the sampling surface.

Results Summary

	Type of Analysis			
Formulation	Microvac structure count million s/cm^2	Passive million s/cm^2	Wipe million s/cm^2	Microvac gravimetric ng/cm^2
0.1% in $CaCO_3$ (normalized to 7 g)	1.2 0.80 - 1.4 0.80 - 1.6	0.87 0.79 - 0.98 0.56 - 1.2	1.9 1.6 - 2.1 1.6 - 2.3	72 60 - 93 0.0022% - 0.0030%
1% in $CaCO_3$ (normalized to 7 g)	7.5 6.2 - 8.4 5.3 - 9.3	7.0 6.0 - 7.8 5.6 - 8.3	17 16 - 18 15 - 19	3,600 870 - 8,800 0.035% - 0.28%
1% replicate in $CaCO_3$ (normalized to 7 g)	7.8 6.3 - 8.8 5.7 - 9.7	NA	NA	NA
3 x 1% in $CaCO_3$ (normalized to 7 g)	9.8 9.0 - 11 7.9 - 11	10 9.7 - 11 9.2 - 11	17 16 - 18 16 - 19	120,000 4,100 - 190,000 0.047% - 2.4%
10% in $CaCO_3$ (normalized to 7 g)	150 140 - 180 120 - 180	130 120 - 140 110 - 150	200 190 - 200 170 - 230	150,000 53,000 - 340,000 1.6% - 13%
1% in Vermiculite (normalized to 7 g)	5.6 4.8 - 5.9 4.2 - 7.1	6.6 5.3 - 7.9 5.0 - 8.4	7.4 7.3 - 7.5 6.5 - 8.4	NA

Normalized Results

(Normalized to 1% asbestos; 7 grams application)

	Type of Analysis			
Formulation	Microvac structure count million s/cm^2	Passive million s/cm^2	Wipe million s/cm^2	Microvac gravimetric ng/cm^2
0.1% in $CaCO_3$	12	8.7	19	720
1% in $CaCO_3$	7.5	7.0	17	3,600
1% replicate in $CaCO_3$	7.8	NA	NA	NA
3 x 1% in $CaCO_3$	9.8	10	17	40,000
10% in $CaCO_3$	15	13	20	15,000
1% in Vermiculite	5.6	6.6	7.4	NA

Gravimetric Analysis - Large Structure Effects

Sample #	Mass of 1st structure	Mass of next 500 structures	1st structure % of total	# needed to reduce to 10%	Projected time required
1A	0.0057 ng	0.047 ng	11	550	8 hrs.
1B	0.014 ng	0.061 ng	19	1,000	15 hrs.
1C	0.024 ng	0.033 ng	42	3,300	50 hrs.
2A	0.051 ng	0.061 ng	46	3,800	57 hrs.
2B	0.046 ng	0.025 ng	65	8,300	120 hrs.
2C	0.45 ng	0.046 ng	91	44,000	660 hrs.
4A	0.08 ng	0.067 ng	54	5,400	81 hrs.
4B	5.5 ng	0.045 ng	99	550,000	8,300 hrs.
4C	0.98 ng	0.15 ng	87	29,000	440 hrs.
5A	1.9 ng	0.062 ng	97	140,000	2,100 hrs.
5B	0.22 ng	0.45 ng	33	2,200	33 hrs.
5C	0.24 ng	0.20 ng	55	5,400	81 hrs.